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Laser induced damage enhancement due to stainless steel deposition on KS-4V and KU1 quartz glasses

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Abstract

LIDAR (light detection and ranging) diagnostic systems for fusion devices will require windows with extremely high transmission in the visible range as a consequence of the use of high power lasers. Within the ITER diagnostics programme, KU1 and KS-4V quartz glasses (OH contents: 800–900 ppm for KU1 and <0.2 ppm for KS-4V) are being examined for such applications. A comparison between the two materials has been carried out in terms of laser induced damage and it was observed that both materials exhibited almost identical behaviour. Laser induced damage enhancement due to stainless steel deposition on both materials has been studied, as well as the possibility of 'in situ' cleaning. Important differences between sputtered and evaporated layers, as well as type of material deposited, were observed.

1. Introduction

LIDAR (light detection and ranging) is one of the important promising diagnostic systems being considered for use in ITER. The system will employ a high intensity laser beam (Ti:Sapphire at about 800 nm with pulses of 1 ns and energy 0.6 J is being considered) which must pass along a channel containing windows and mirrors. Due to the high power of the laser beam the windows must have extremely high transmission in the visible range to avoid damage (laser power density at the windows will of course depend on focusing). Within the ITER diagnostics programme, KU1 and KS-4V quartz glasses (OH contents: 800–900 ppm for KU1 and <0.2 ppm for KS-4V) provided by the Russian Federation are being examined for such applications [1].

KU1 and KS-4V are known to be highly radiation resistant [2–4]. In addition, work on laser damage during irradiation showed KU1 to be excellent in terms of dose and dose rate effects [5]. However it was later shown that the growth of a thin (5 nm) conductive layer of gold on KU1 dramatically decreased the intensity threshold to produce laser damage by several orders of magnitude [6]. Following this, more realistic stainless steel layers from 5 to 200 nm were deposited on KU1 by evaporation and sputtering. Important differences were observed between the two deposition methods. A comparison was then made between KU1 and KS-4V. Both materials exhibit almost identical behaviour in terms of laser induced damage. The possibility of 'in situ' laser cleaning was studied for thin layers of stainless steel deposited by both evaporation and sputtering.

2. Experimental procedure

Samples of KU1 and KS-4V, 16 mm diameter and 1 mm thick were optically polished at the same time to ensure as near as possible identical surface conditions. To simulate the effect of contamination which may be deposited with time on the vacuum face of optical windows of ITER or similar fusion devices, thin stainless steel (310 SS type) layers were deposited by 1.4 keV Ar sputtering onto the optically polished KU1 and KS-4V

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quartz glass samples. A surface profiler DEKTAK was used to determine the thickness of the layers. In the initial tests stainless steel was also deposited on KU1 by evaporation. Before and after deposition, the samples were characterized for optical transmission, and SEM X-ray analysis was carried out to determine the elements in the deposited layers.

To study damage a Q-switched Nd:YAG laser was used (1064 nm, 7 ns pulse of maximum energy 330 mJ). The laser beam direction was normal to the surface of the sample, and was focused using a 50 mm focal length lens, in the exit surface layer of the sample. The focal spot size was 30 μm. The samples were mounted in a holder which could be moved vertically and horizontally in the focal plane of the lens, and the position determined to about 0.1 mm. To determine damage probability at different laser power densities, each sample was laser irradiated at a different point for each pulse at a determined power. After each laser pulse, the sample was moved horizontally in the focal plane of the lens from between 0.5 mm to more than 1 mm, depending on the extension of the damage, so that a new material zone was used for each laser pulse. When necessary the sample could be moved vertically. Measurements were carried out laser irradiating for nine different power densities (from 1×10^{14} to 6.7×10^{16} W/m^2) and for each power density at 25 different positions. Laser damage was monitored visually by light emission and also by a clearly audible noise resembling a small explosion. To check the damage site the sample was observed in an optical microscope where craters or cracks from 0.1 to 1 mm were observed. Damage was not visible in regions where neither light nor sound emission were observed.

The possibility of 'in situ' laser cleaning was assessed for thin layers of stainless steel deposited by both evaporation and sputtering by examining the regions which had been illuminated with laser pulses below the damage threshold.

3. Results

Important differences were observed between the two deposition methods. In the case of evaporation the layers were highly reflecting and electrically conductive from the beginning, even for very thin layers. However in the case of sputtering, layers up to approximately 30 nm showed high electrical resistance (>200 GΩ) and low reflectivity, indicating that the deposited material was an insulator rather than a conductor. On increasing thickness the layer became reflecting and conductive indicating a change to a metallic phase. The adhesion of the evaporated layers was poor and were easily scratched or removed by wiping. In marked contrast the sputtered layers were extremely difficult to remove, a physical/chemical reaction must take place between the energetic

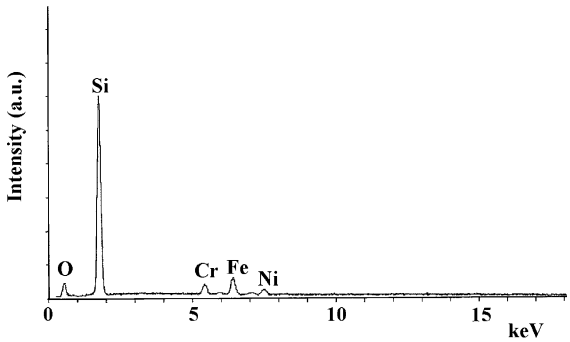

Fig. 1. X-ray SEM analysis for 200 nm stainless steel sputter deposition on KU1 quartz glass.

atoms sputtered from the stainless steel target and the KU1 quartz glass surface, highly enhancing the adhesion. SEM X-ray analysis indicated that the proportion of elements present was as expected. This is shown in Fig. 1 for a KU1 sample where Fe, Cr, and Ni are present in the deposited layer (310 SS: Fe/Cr25/Ni20). The silicon and oxygen peaks correspond to the silica substrate.

Fig. 2 shows the transmission spectra for optically polished KU1 and KS-4V quartz glasses. The main differences are due to the large infrared OH absorption bands for KU1. The figure also shows the transmission with a layer of ~70 nm of sputtered stainless steel. One can see that at the laser wavelength (1064 nm) the transmission reduces from about 93% to 88% due to deposition.

The samples were then examined for laser damage. For the sputtered layers, the damage threshold gradually decreased with increasing layer thickness, and by 20 nm

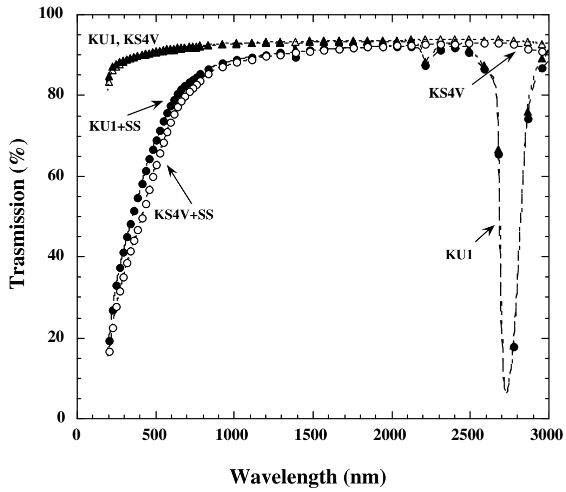

Fig. 2. Optical transmission spectra for KU1 and KS-4V before and after 70 nm stainless steel deposition by sputtering.

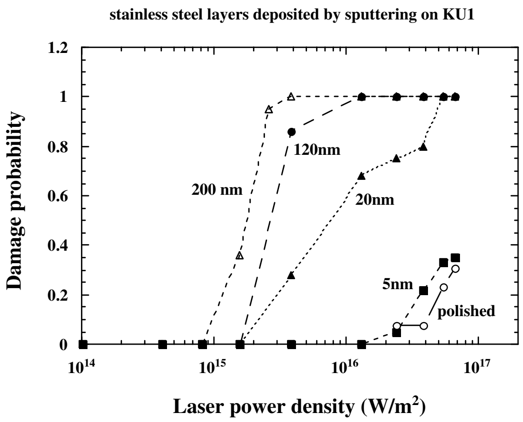

Fig. 3. Damage probability as a function of laser power density for different stainless steel sputtered KU1 quartz glass samples: (○) polished, (■) 5 nm stainless steel layer, (▲) 20 nm stainless steel layer, (●) 120 nm stainless steel layer, (△) 200 nm stainless steel layer.

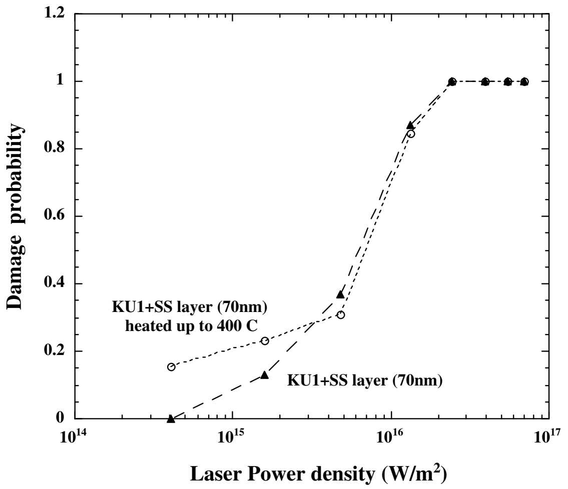

Fig. 5. Damage probability as a function of laser power density for KU1 stainless steel sputtered (▲) and sputtered and heated in vacuum at 400 °C during one hour (○).

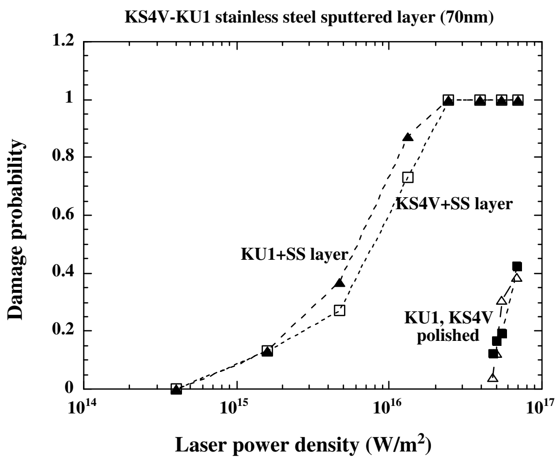

Fig. 4. Comparison of damage probability as a function of laser power density for KU1 and KS-4V. Sputtered stainless steel layer 70 nm.

was one order of magnitude lower. Fig. 3 gives the damage probability for KU1 as a function of laser power density for four metallized samples (5, 20, 120, 200 nm sputtered stainless steel).

Fig. 4 shows the damage probability for KU1 and KS-4V, optically polished and with a 70 nm sputtered stainless steel layer. There is clearly no difference between KU1 and KS-4V with respect to their damage resistance.

After heating in vacuum at 400 °C a very slight increase of damage probability was observed at the lower laser intensities (Fig. 5). In contrast, no effect on the damage threshold was observed for the evaporated layers because even at low laser powers these layers were readily removed from the KU1 surface, probably by re-evaporation.

To assess possible 'in situ' laser cleaning, the regions which had been illuminated with laser pulses below the damage threshold were examined. It was observed that in the case of stainless steel deposited by sputtering it was impossible to remove the deposit without damaging the KU1. However, as indicated above, the evaporated stainless steel was easily removed from the KU1 substrate by laser pulses with intensities below the damage threshold.

4. Discussion

KU1 and OH free KS-4V show almost identical damage behaviour (Fig. 4) indicating that OH groups do not play an important role in the laser damage process. Similar results were obtained in earlier work on laser damage of quartz glass where almost no dependency with the OH content was observed [7].

As may be seen in Figs. 3 and 4, KU1 and KS-4V exhibit high resistance to laser damage, but the damage threshold slowly decreases with sputtered steel deposition thickness. This is in marked contrast to sputtered gold where the damage threshold dramatically decreased even for a 5 nm layer [6].

The sputtered gold and evaporated steel layers were very similar in several aspects; they were both electrically conducting even for very thin (5 nm) layers, they both showed high reflection, and both could be removed by laser pulses without damage, though in the case of gold the margin between removal or damage was very small.

In transmission windows laser damage occurs at the exit interface between the insulator and the contaminating layer where absorption and reflection produce a localized hot spot with plasma and UV emission, inducing an increase in the insulator electrical conductivity [8,9]. However from this general description it is difficult to predict the effect of a particular contaminating layer, but damage clearly becomes more important when the stainless steel sputtered layer is electrically conducting and reflecting (from Figs. 3 and 5). The results highlight the large differences between the effects of evaporation or sputtering, and type of sputtered material, iron or gold.

Laser cleaning is being considered as a way to reduce contamination layers on the vacuum face in ITER diagnostic windows. This has been successful for sputtered gold and evaporated carbon [6,10]. In the present experiments it was easy to remove evaporated steel. However laser cleaning was not possible for sputter deposited steel without damaging the samples, possibly related to the enhanced adhesion. Finally the effect of temperature on laser damage was addressed, as window temperatures in ITER could be high due to nuclear heating. After heating in vacuum at 400 °C for one hour, the sputtered steel layer on the silica suffers oxygen reduction causing an increase in the reflectivity and electrical conductivity. This slightly increased the probability of laser damage, in agreement with the observations above.

5. Conclusions

Both KU1 and KS-4V are very resistant to laser damage, and their behaviour is almost identical. However for both materials sputtered stainless steel deposition noticeably enhances laser damage probability. Laser damage probability is further enhanced on heating in vacuum. While laser cleaning of evaporated steel and carbon, and sputtered gold is feasible, sputtered steel could not be removed. In view of the very different results recorded for sputtering and evaporation of steel, gold, and carbon, laser damage studies should be extended for other materials such as tungsten and beryllium expected in ITER.

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